WRDC-TR-89-4102

A CHARACTERIZATION OF DEXTER HYSOL'S MODIFIED ACETYLENE-TERMINATED BISPHENOL-A PREPREG SYSTEM: XAS/AF-8

PERRY R. WAGNER
STRUCTURAL MATERIALS BRANCH
NONMETALLIC MATERIALS DIVISION

JULY 1989

FINAL REPORT FOR PERIOD NOVEMBER 1987 - JULY 1988

APPROVED FOR PUBLIC RELEASE -- DISTRIBUTION IS UNLIMITED

MATERIALS LABORATORY
WRIGHT RESEARCH & DEVELOPMENT CENTER
AIR FORCE SYSTEMS COMMAND
WRIGHT-PATTERSON AIR FORCE BASE, OHIO 45433-6533

UNCLASSIFIED
SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

Form Approved
OMB No. 0704-0188

1a. REPORT SECURITY CLASSIFICATION Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT Approved for public release; Distribution is umlimited
2b. DECLASSIFICATION/DOWNGRADING SCHEDULE	
4. PERFORMING ORGANIZATION REPORT NUMBER(S) WRDC-TR-89-4102	5. MONITORING ORGANIZATION REPORT NUMBER(S)
6a. NAME OF PERFORMING ORGANIZATION Materials Laboratory WRDC/AFSC 6b. OFFICE SYMBOL (If applicable) WRDC/MLBC	7a. NAME OF MONITORING ORGANIZATION
6c. ADDRESS (City, State, and ZIP Code) WRDC/MLBC WPAFB, OH 45433-6533	7b. ADDRESS (City, State, and ZIP Code)
8a. NAME OF FUNDING/SPONSORING ORGANIZATION 8b. OFFICE SYMBOL (If applicable)	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
8c. ADDRESS (City, State, and ZIP Code)	10. SOURCE OF FUNDING NUMBERS PROGRAM ELEMENT NO. 6210F; PROJECT NO. 2419; TASK NO 03; WORK UNIT ACCESSION NO. 10

11. TITLE (Include Security Classification)
"A Characterization of Dexter Hysol's Modified Acetylene-Terminated Bisphenol-A Prepreg System: XAS/AF-8"

12. PERSONAL AUTHOR(S)
Wagner, Perry R.

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM Nov 87 TO Jul 88	1 Jul 89	41

16. SUPPLEMENTARY NOTATION

17. COSATI CODES			18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number)
FIELD	GROUP	SUB-GROUP	prepreg, acetylene-terminated resin, advanced composites, mechanical properties, ductility.

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The objective of this research effort is to characterize an advanced composite prepreg system. The material under consideration is Dexter Hysol's modified acetylene-terminated bisphenol-A resin on continuous XAS-type graphite fibers. Hysol asserts that this modified compsoite system retains the 400-425°F use temperature associated with state-of-the-art acetylene-terminated resin prepreg systems while achieveing a 64% increase in Mode I fractur toughness. Our investigation shows that although the ductility was improved, other mechanical properties were severely degraded when test coupons were evaluated at 350°F and saturated with water.

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT	21. ABSTRACT SECURITY CLASSIFICATION
☒ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS	Unclassified

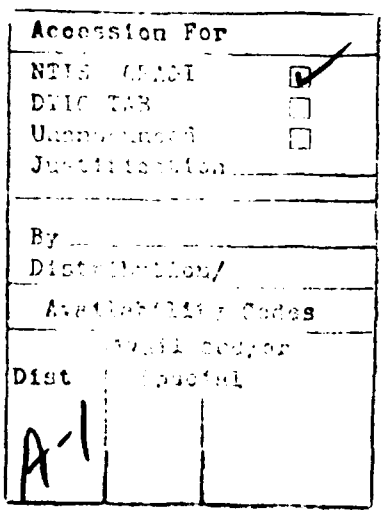
22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code)	22c. OFFICE SYMBOL
Perry R. Wagner	(513) 255-9058	WRDC/MLBC

DD Form 1473, JUN 86 *Previous editions are obsolete.* SECURITY CLASSIFICATION OF THIS PAGE

UNCLASSIFIED

FOREWORD

This technical report is the culmination of a 9 month research effort extending from November 1987 to July 1988. The author is Perry R. Wagner, whose background is the mechanical testing of composites. The work was performed at the Materials Laboratory, Wright Research & Development Center located at Wright-Patterson AFB, Ohio.



ACKNOWLEDGEMENTS

I would like to thank the following individuals, all of whom made invaluable contributions during this research effort: Brian Rice, Ron Cornwell, Chuck Fowler, Ken Lindsay, Bill Price, and Bill Ragland. The aforementioned are members of the University of Dayton Research Institute on-site contract at Wright-Patterson AFB, Ohio.

TABLE OF CONTENTS

LIST OF FIGURES

FIGURE PAGE

LIST OF TABLES

TABLE PAGE

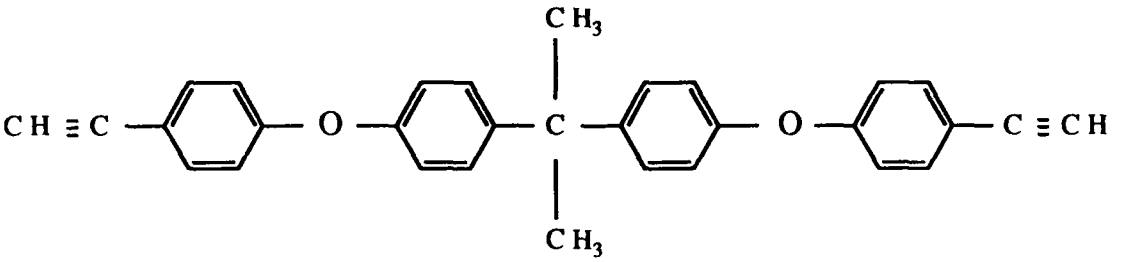
I. INTRODUCTION

The WRDC Materials Laboratory has an ongoing effort to evaluate new materials which have demonstrated a potential use on future Air Force weapon systems. Typically, a modified composite prepreg system, which has been developed under Materials Laboratory contractual efforts, warrants further in-house investigation. Such material systems are reserved for new hires desiring experience in composites fabrication and characterization. It was under these circumstances that this research investigation was initiated. The data reported here is not design data. On the contrary, this effort can be classified as an initial material screening.

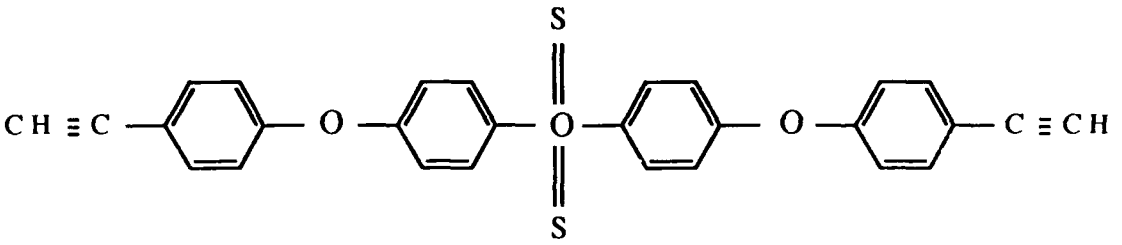
The material to be examined is a composite prepreg system consisting of Hysol-Grafil continuous graphite fibers impregnated with a modified ("toughened") acetylene-terminated bisphenol-A resin. The system was purported to have a 400°-425°F use temperature and a Mode I fracture toughness value higher than that of previous resin prepregs.

II. BACKGROUND

With the inception and use of graphite/epoxy technology, the design of many structural applications has changed significantly. Specifically, the Air Force has changed many of their load-bearing aircraft components from aluminum alloys to graphite/epoxy. The major drawback to this composite system, however, is its hygroscopic nature.[1] The polar groups within the epoxy matrix attract water which then serves as a plasticizer, lowering the glass transition temperature (Tg) of the matrix and, thus, limiting the use temperature of the composite system and structure. It was this problem that encouraged the development of resins with better "hot/wet" characteristics--resins whose properties do not degrade as rapidly as the epoxy resins upon exposure to higher temperatures and humidities. Bismaleimide, polyimide, and AT imide resins were developed to solve this problem. AT (acetylene terminated) molecules were first conceived in the 1960's. They can sustain use temperatures of 500°-600°F for limited periods of time. In addition, AT resins were touted as being practically insensitive to moisture and easily processable for organic matrix structural composite applications. Their hydrophobic nature was indeed a reality, but their ease in processing was not confirmed. These AT resins were brittle. And, their cost was nearly $200/lb. Thus, the Materials Laboratory again sponsored contractual programs to augment the structure of the AT molecule in hopes of improving its processing characteristics and lowering its cost. The resulting ATB (acetylene-terminated bisphenol-A) and ATS (acetylene terminated sulfone) molecules are shown in Figure 1. Contracts were established with Dexter Hysol and American Cyanamid in 1985 to "toughen" the ATB and ATS resins. The formulated AT resins were scaled up and applied to graphite fibers. The resulting prepreg was shipped in 5 pound batches to various airframe corporations and to the Materials Laboratory.[2]



ATB



ATS

Figure 1 Acetylene-Terminated Bisphenol-A and Sulfone Structures.

Thus, it was the 5 pound batch of ATB prepreg which was sent from Hysol to the Materials Laboratory that was characterized under this research effort. Hysol's trademark names for the ATB-based resin systems are designated AF-1 through AF-10. Their trademark names for ATS-based systems are AF-11 through AF-20. AF-4 and AF-8 were chosen by Hysol as being two of the better ATB-based resin formulations. The XAS/AF-8 system was examined during this research effort. Hysol chose the Hysol-Grafil XAS fiber as the reinforcement based on its interface characteristics with the resin.

Limited time and materials restricted the performance of an extensive characterization. However, during this project, a reasonable amount of physical and mechanical data was amassed. One of the goals of this research was to focus on the toughness and interlaminar shear strength properties of the XAS/AF-8 system. However, other important mechanical and physical data were gathered in order that the experience base associated with the AT resins would be broadened.

III. EXPERIMENTAL PROCEDURES

A. Prepreg Characterization

The 5 pound batch of prepreg was received from Hysol on 12 May 1987 and was placed in cold storage. Prior to cutting up the prepreg into sections for lay-up, several physical property tests were performed as a matter of course. Samples were sectioned from the Hysol prepreg and were tested in a DuPont 910 differential scanning calorimeter (DSC) with an Omnitherm Controller. The experiment was run from room temperature to 400°C in nitrogen at a ramp rate of 10°C/min. The DSC run was performed to gain insight into the material's thermal properties such as the heat of reaction, the temperature at which the exotherm begins and the maximum exotherm temperature.

On a replicate section of Hysol prepreg, a thermogravimetric analysis (TGA) was performed using a DuPont 951 model with an Omnitherm Controller. Similar to the DSC, the TGA was operated from room temperature to 400°C in nitrogen at a ramp rate of 10°C/min. The figures of merit with respect to TGA are percent residue [(initial weight-final weight)/initial weight] and temperature at onset of volatilization.

In order to obtain a basic understanding of the rheological behavior of the Hysol material, two specimens were run through an RMS 7200 rheological characterization unit. The first run was made using a 10-ply, unidirectional prepreg sample (1.25" x 0.47" x 0.063"). The second sample (1.95" x 0.50" x 0.075") was a 12-ply, unidirectional cured composite (its cure cycle being the same as was used for all the consolidation runs). Both samples were cycled in the RMS from room temperature to 350°C and back to room temperature at a rate of 2°C/min. Both tests were performed in nitrogen. Each of the two experiments was performed at three different frequencies (0.16 Hz, 1.6 Hz, and 15.92 Hz). The curves generated from these tests provide a good deal of information about the material, most importantly its glass transition temperature. The storage modulus, loss modulus, and the ratio of these two terms were recorded at each frequency as a function of temperature. The as-cured sample was run through the RMS to see if the material had fully cured.

B. Panel Fabrication

A total of six panels were cut from the prepreg roll, laid up into various orientations, bagged, and finally cured in a Thermal Equipment Autoclave (model 8397). A schematic describing the bagging procedure is shown in Figure 2. The cure cycle and post-cure cycles (Table 1) were used for all six panels.

Table 1 Manufacturer's Cure And Post-cure Cycles for XAS/AF-8 Prepreg.

XAS/AF-8 Cure Cycle

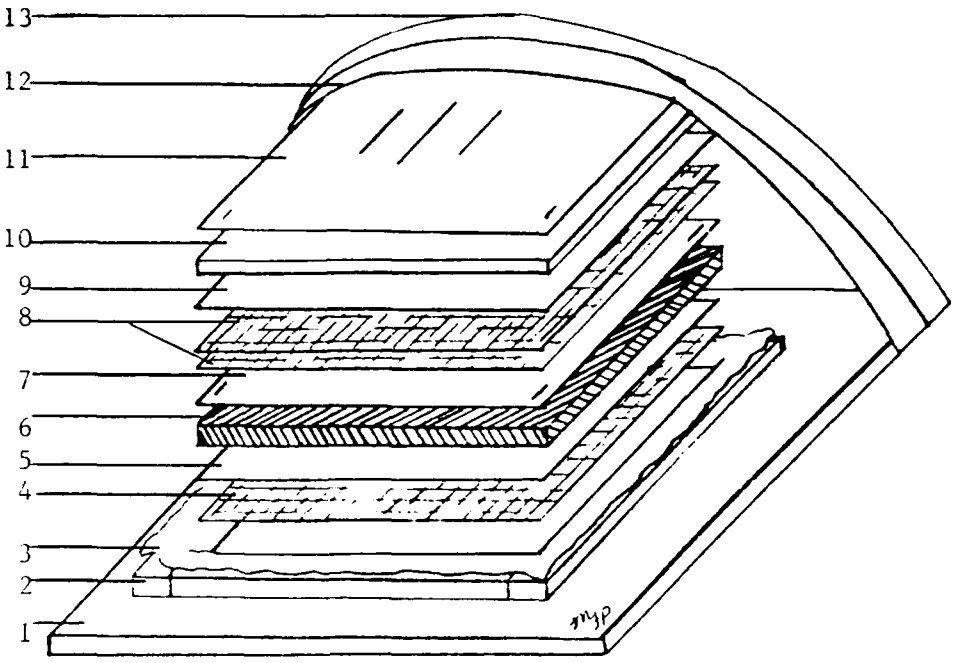
Pressurize to 100 psi
Heat-up to 235°F at 2-3°F/minute.
Hold at 250°F for 30 minutes.
Heat-up to 355°F at 2-3°F/minute.
Hold at 355°F for 300 minutes.
Cool down at 1-2°F/minute.

XAS/AF-8 Post-Cure Cycle

Heat-up to 511°F at 3°F/minute.
Hold at 511°F for 240 minutes.
Cool down at 3°F/minute

C. Physical Testing

Every panel was exposed to a series of physical evaluations in order to check for noticeable delaminations and/or voids and to determine the overall laminate density and fiber volume. C-scans of all six panels were performed on a Testech Ultrasonic Immersion system. From each of the panels, a section (approximately 1 square inch) was reserved for photomicrographs, density, and fiber volume coupons. The photomicrograph specimen was mounted in epoxy and photographed at various magnifications (i.e., 150X and 300X) with an Olympus camera. Other specimens from the section were weighed both in water and in air to obtain the density. These same specimens were then placed in a heated sulfuric acid bath for several hours. The residual fibers were weighed and the fiber, matrix, and void percent content by volume were calculated. Fiber and resin specific gravity values were obtained from the manufacturer to be used in these calculations.



SPECIFICATIONS

1- MOLD RELEASE ON CAUL PLATE
2- CORK DAM
3- POUROUS TEFLON COATED GLASS FABRIC; SIDES ARE FOLDED AROUND AND TAPED WITH HIGH TEMPERATURE TAPE TO FORM A PACKAGE WHICH INCLUDES SPECIFICATION NUMBERS, 4 THROUGH 11.
4- BLEEDER CLOTH (1 for 12"X12", 2 FOR 6"X9")
5- POUROUS TEFLON COATED GLASS FABRIC
6- LAMINATE
7- NONPOUROUS TEFLON COATED GLASS FABRIC
8- BLEEDER CLOTH
9- POUROUS TEFLON COATED GLASS FABRIC
10- CAUL PLATE
11- NONPOUROUS TEFLON COATED GLASS FABRIC
12- GLASS FABRIC
13- VACUUM BAG

Figure 2 Bagging Schematic For Consolidation of Panels.

D. Mechanical Testing

The six panels were machined into coupons which were then subjected to the test matrix shown in Table 2. The number of specimens performed under a given test and given condition is shown in brackets. This matrix provides for a rudimentary mechanical analysis of the ATB material. All of the tests are static in nature. The mechanical properties obtained from this test matrix can be classified into two groups: those that fill the compliance matrix and those that describe the fracture behavior of the material. Tests which supply information to the compliance matrix are 0°-4 point flex, 0°-3 point flex, 0°-4 point shear, short beam shear, 90°-4 point flex, 0°-tension and ±45°-tension. The Mode I, Mode II, and edge delamination tests provide insight into the composite's fracture characteristics.

It is rare that a mechanical test (even one that is performed meticulously under the strictest standards) provides a "true", inherent material property. In the test matrix of Table 2, only the 0°-tension and ±45°-tension tests offer true material properties. The former measures the longitudinal strength and modulus, while the latter measures the in-plane shear strength and modulus. The flex and shear coupons, on the other hand, are exposed to such biaxial (and even triaxial) states of stress that the resulting data fall under severe scrutiny from the research community.[3,4] The test data are further confused when the test coupons are exposed to humidity and/or tested at elevated temperatures.[5]

Table 2 Mechanical Test Matrix Used To Analyze XAS/AF-8.

Test Temperature	74	250	350	74	250	350
Aging Conditions	Dry	Dry	Dry	Wet	Wet	Wet
Mechanical Test						
0°-4 Point Flex	[5]	[5]	[5]	[5]	[5]	[5]
0°-3 Point Flex	[5]	[5]	[5]	[5]	[5]	[5]
0°-4 Point Shear	[5]	[5]	[5]	[5]	[5]	[5]
Short Beam Shear	[5]	[5]	[5]	[5]	[5]	[5]
90°-4 Point Flex	[5]			[5]		
Mode II	[5]					
0°-Tension	[5]					
+/-45°-Tension	[5]		[5]	[5]		[5]
Edge Delamination	[5]					
Mode I	[5]					

Test	Test Coupon Orientation	Nominal Coupon Geometry Length (in.)	Width (in.)	Thickness (in.)	Test Standard Used	Instron Model On Which Tested	Tabbing Required	Type of Strain Measuring Device	Span-to Depth Ratio
0°-4 Point Flex	[0°]12	3.0	0.50	0.07	ASTM D790-81	1123	No	Crosshead	32/1
0°-3 Point Flex	[0°]12	3.0	0.50	0.07	ASTM D790-81	8086	No	Crosshead	32/1
0°-4 Point Shear	[0°]12	3.0	0.50	0.07	ASTM D790-81	1123	No	Crosshead	16/1
Short Beam Shear	[0°]12	1.0	0.25	0.07	ASTM D2344-84	8086	No	Crosshead	4/ 1
90°-4 Point Flex	[90°]12	3.0	0.50	0.07	ASTM D790-81	1123	No	[1]	32/1
Mode II	[0°]24	6.0	1.00	0.07	D30	1123	No	Crosshead	span = 4"
0°-Tension	[0°]12	9.0	1.00	0.07	ASTM D3039-76(82)	1115	Yes	Extensometer	N/A
+/-45°-Tension	[+/-45°]2s	9.0	1.00	0.05	ASTM D3518-76(82)	1115	Yes	[2]	N/A
Edge Delamination	[+/-/-/+30°/(90°)2]s	9.0	1.00	0.07	D30	1115	Yes	Extensometer	N/A
Mode I	[0°]24	9.0	1.00	0.14	D30	1115	Yes	Crosshead	N/A

[1] BLH type FAE-18-12-S6HL strain gage

[2] BLH type FAET-12D-12-S6HL strain gage

Table 3 Mechanical Tests And pertinent Test Data.

previous caveats noted, the tests in Table 2 will be further discussed with respect to other key test parameters. Table 3 provides a comprehensive overview of the important data associated with each mechanical test. Miscellaneous information related to Table 3 is noted below.

The 0°-4 point flex and 0°-3 point flex tests were performed to measure an estimate of the properties parallel to the fiber. The 90°-4 point flex test investigated properties perpendicular to the fiber axis. The 0°-4 point shear and short beam shear tests provide an estimate of the interlaminar shear properties.

There are no standardized methods to date for the Mode I, Mode II, and edge delamination tests. However, the ASTM D30 committee is presently reviewing these three tests. All are very well documented and are in the round robin stage. The edge delamination test provides insight into the Mode I fracture toughness of a composite material. The coupon's geometry and loading conditions are identical to those of the 0°-tension coupon. The sides of the edge delamination coupon are polished and/or marked with a white paint so that ply delaminations can be easily observed. The key parameters for this test are the initial modulus, stress at delamination, ultimate strength and ultimate strain-to-failure. The Mode I test coupons were machined from a 12-ply unidirectional laminate. The last 1" of the panel has a Teflon release ply positioned halfway through the panel thickness. Aluminum tabs were adhesively bonded to the cracked region of the coupon and then pulled apart. The Mode I test measures the Mode I-type fracture toughness (G_{Ic}). Figure 3 shows a schematic of the Mode I test coupon.

The Mode II test coupons originated from the same panel from which the Mode I coupons were machined. And, thus, the Mode II test coupons have the same "starter crack." However, the Mode II coupons were tested in a 3-point flex mode with a span of 4". As the crosshead is engaged, the "starter crack" lengthens. From the loads and lengths recorded, a Mode II fracture toughness term is generated.

It is of particular interest with polymer matrix composites to understand how they will survive in hot/wet environments. Table 2 shows that various coupons were moisture-aged prior to testing at a range of temperatures. Three test temperatures were chosen (74°F, 250°F and 350°F) in order that the material's high-temperature behavior be sufficiently examined. A specimen tested under "wet" conditions is one that has been fully saturated with water. Individual specimens were

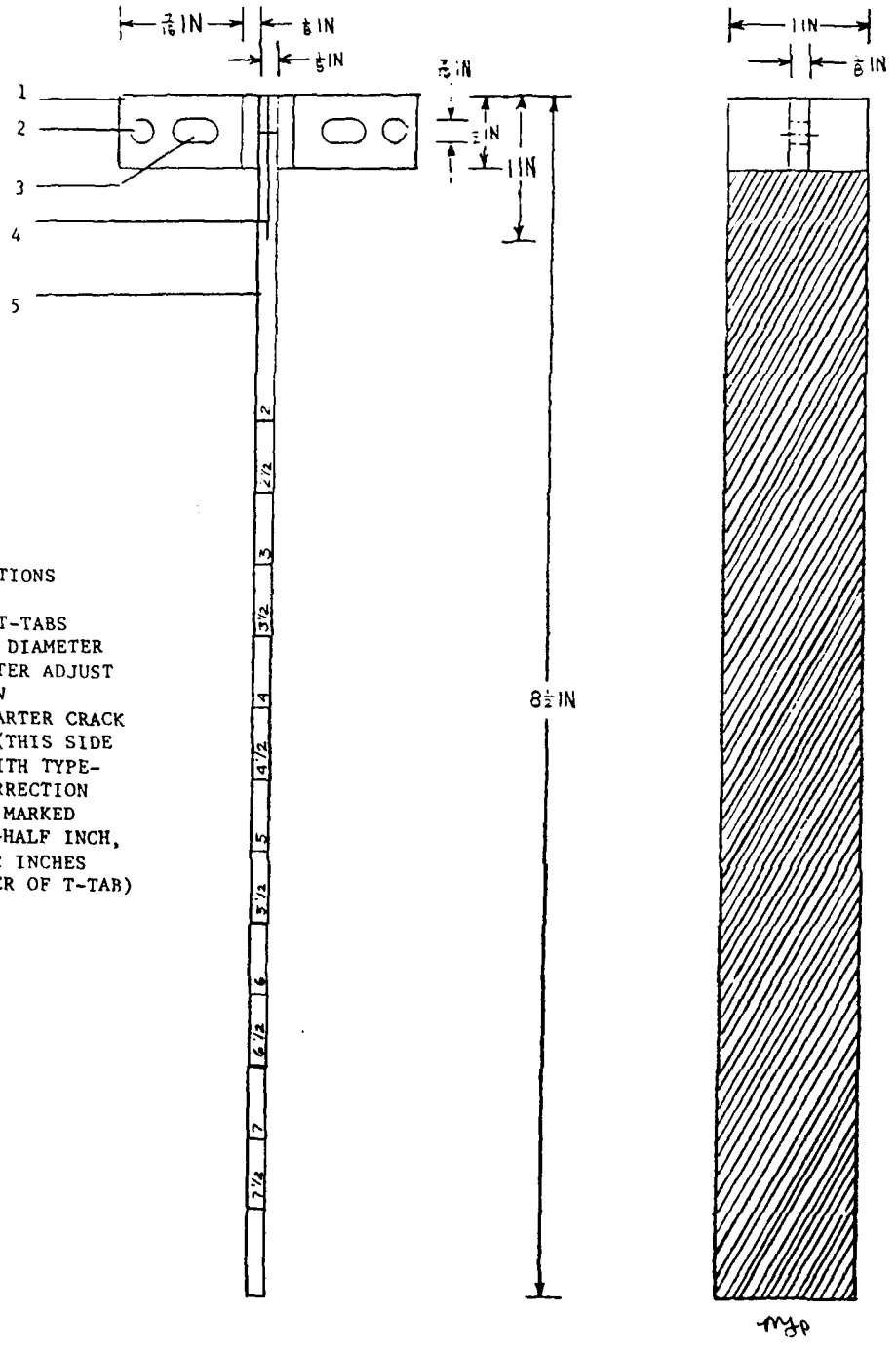


Figure 3 Schematic of the Mode I Test Coupon.

saturated by placing them in a beaker of water in an oven whose temperature was set at 160°F. All of the wet specimens were moisture-aged in three separate batches for convenience. Samples from each batch were weighed periodically and after a sufficient amount of time had elapsed (when the weight increase was observed to level off), the specimens were removed for mechanical testing. Mechanical testing of moisture-aged specimens at high temperatures requires that the saturated specimen remain at temperature no longer than 5 to 10 minutes. Any exposure beyond this time will drive the water from the specimen.

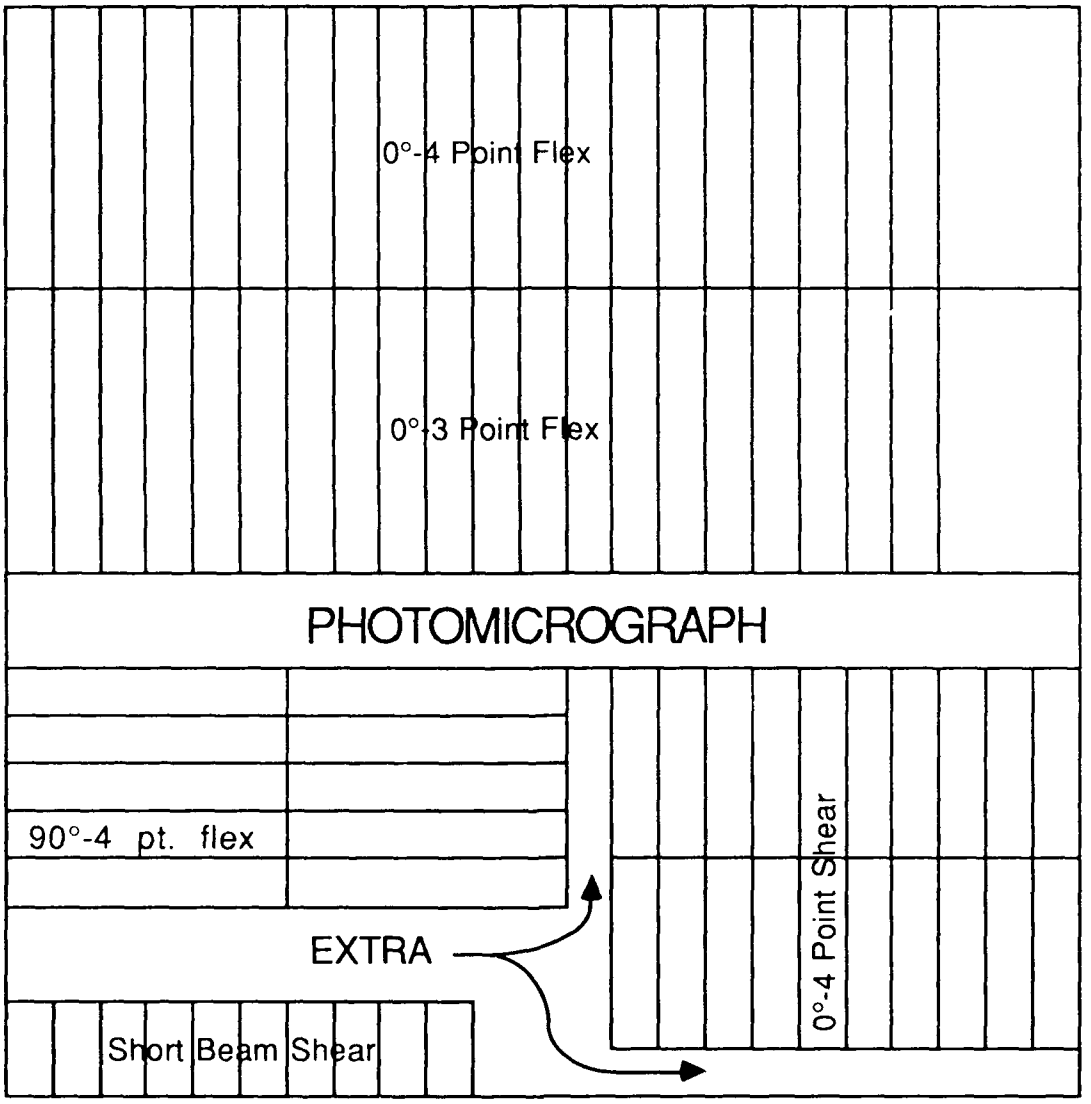


Figure 4 Panel #1: Cut-out Schematic of Flex and Shear Specimens.

A cutting-plan schematic was made for each panel so that specimen machining would be straightforward and efficient. Unfortunately, randomization of specimen layout was not feasible as this would make the task of sectioning the panels inefficient. The cutting schematics of each of the six panels is shown in Figures 4 through 8. These plans were adhered to as rigorously as possible.

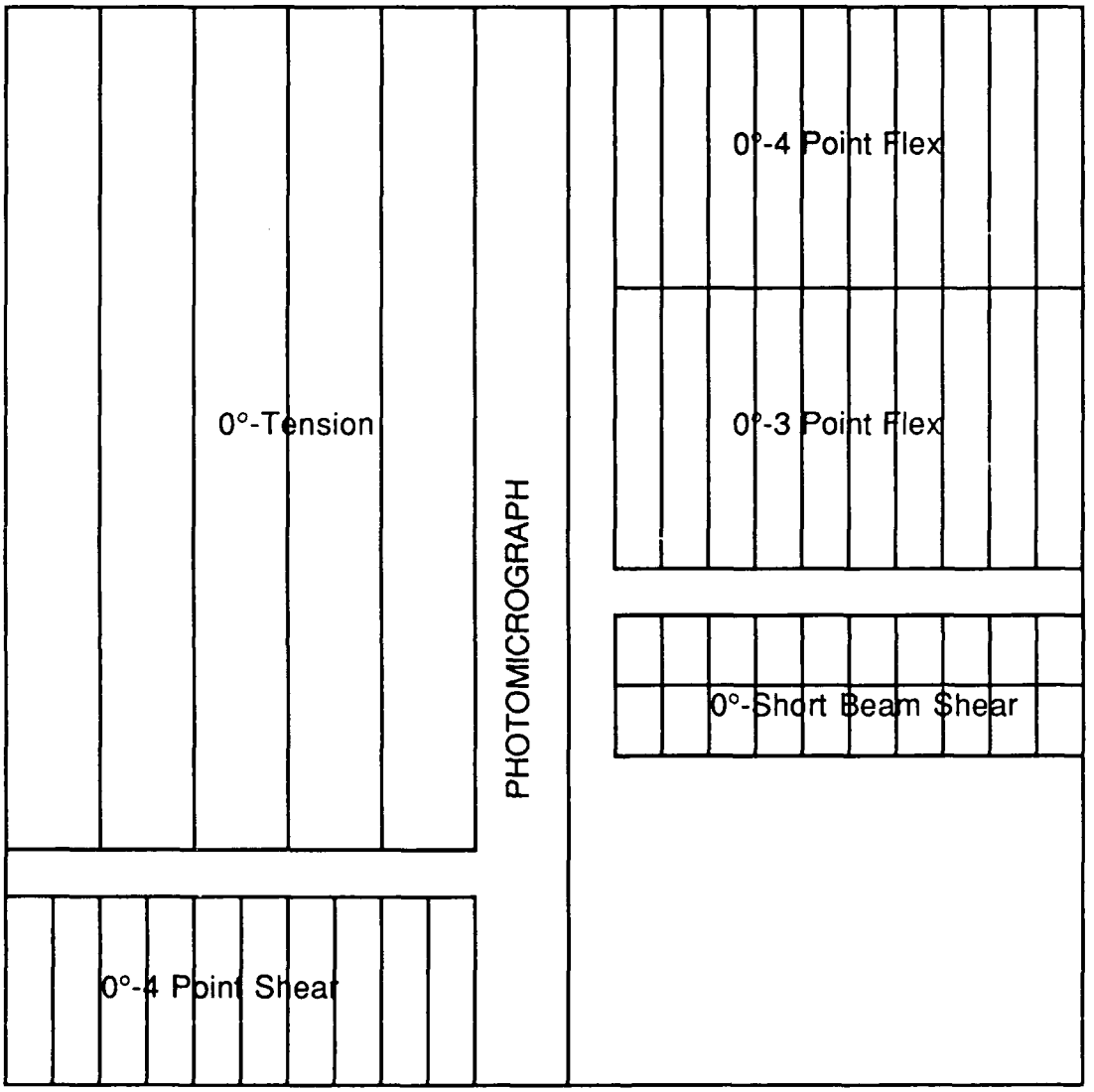


Figure 5 Panel #2: Cut-out Schematic of Tension, Flex and Shear Specimens.

The methods for data reduction are given in the ASTM standards. They were followed as prescribed. For the Mode I, Mode II and Edge Delamination tests, the data reduction schemes specified by the latest round robin version were followed.

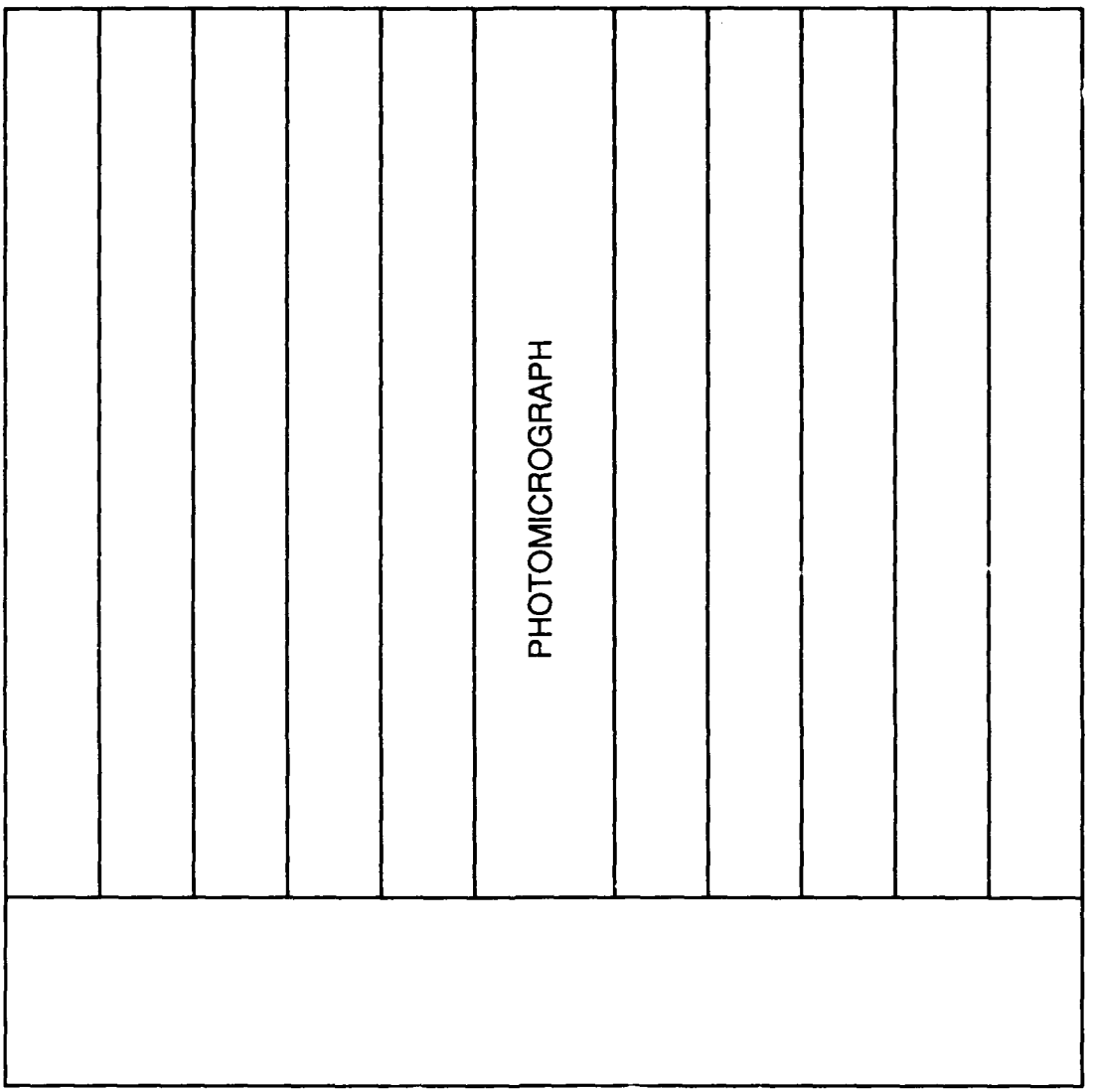


Figure 6 Panels #3,6: Cut-out Schematic of In-plane Shear Specimens.

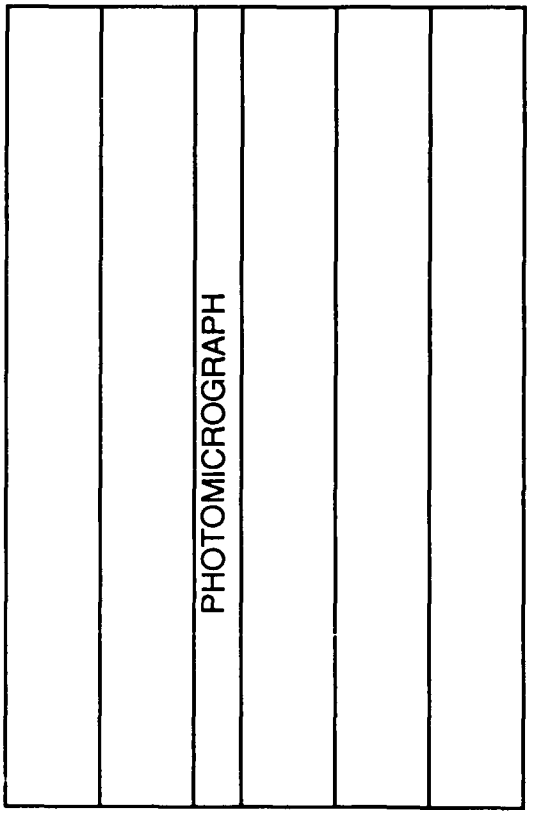


Figure 7 Panel #4: Cut-out Schematic of Edge Delamination Specimens.

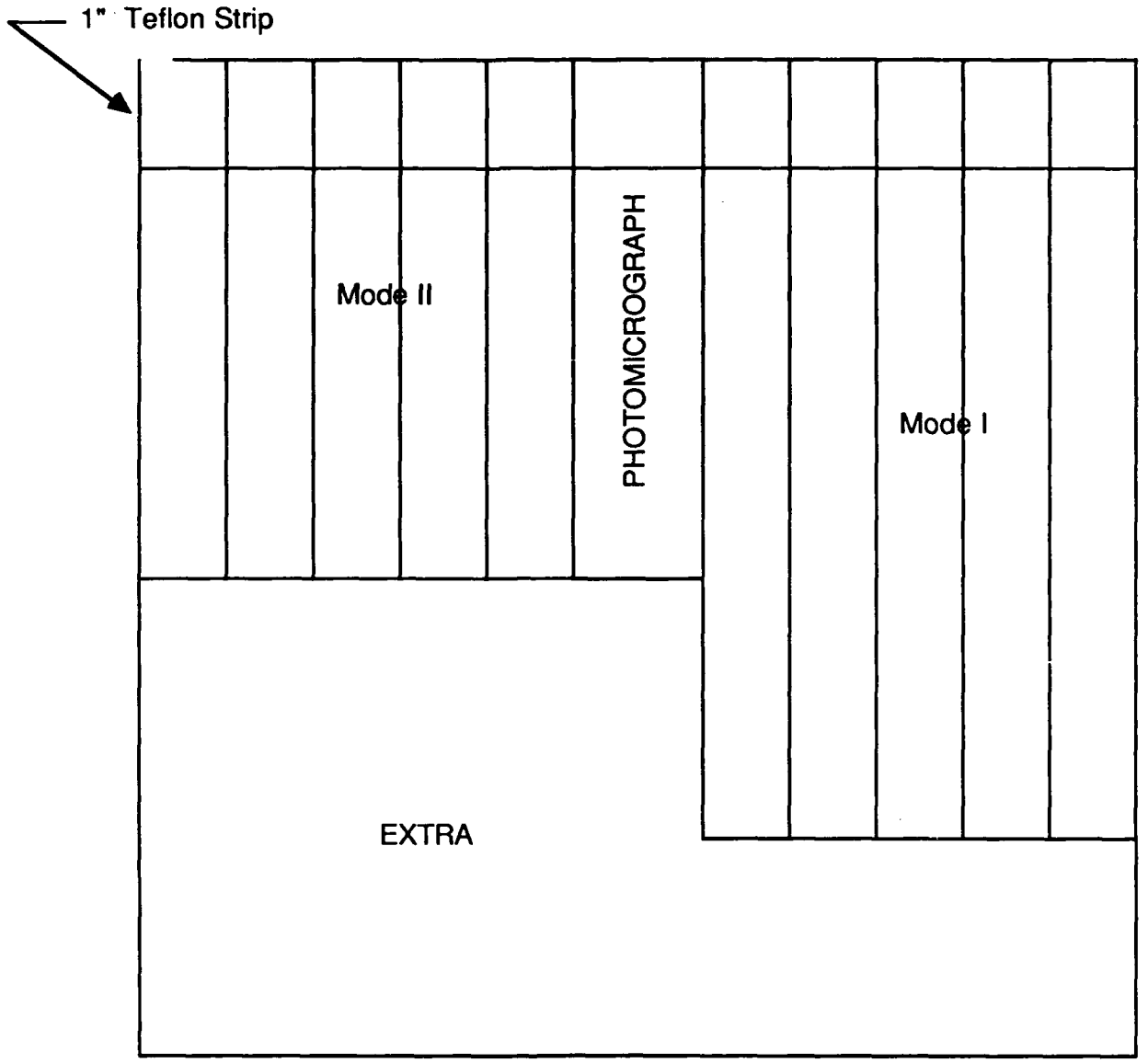


Figure 8 Panel #5: Cut-out Schematic of Mode I and Mode II Specimens.

IV. EXPERIMENTAL RESULTS

A. Physical Properties Data

As the prepreg was being laid-up, it was noticed that it did not have much tack. In addition, it appeared non-homogeneous in places (black in spots and shiny in others). Figures 9 through 12 illustrate the results of the DSC, TGA and rheometrics data. Figure 9 shows the heat of reaction for the ATB prepreg to be 28.5 mcal/mg. The exotherm began at 210°C and peaked at approximately 255°C. Figure 10 shows the onset of volatilization to begin at 169°C. The residual weight percent at 400°C is 94.9%. The onset of gelation occurs at approximately 225°C. (Figure 11: RDS of prepreg section.) The RDS of the cured laminate section (Figure 12) shows the Tg to read roughly 295°C with a torsional frequency equal to 1.6 Hz and a torsional strain of 0.08%.

The ultrasonic c-scans of the six panels appeared to have no delaminations or voids; no anomalies were noticed. The photomicrographs taken from the panels at 150x and 300x displayed no unusually large voids or resin-rich areas. Representative photographs from panel #5 are displayed in Figure 13. Density and fiber volume data for all panels are listed in Table 4. Two specimens from panel #5 (5.a and 5.b) were run as a check because the calculated percent fiber volume was lower than that of any other panel. Both fiber volume calculations from panel #5 support the notion that this panel is resin-rich, despite the fact that the panel's photomicrographs appear normal.

B. Mechanical Data

Table 5 is a summary sheet depicting the pertinent mechanical data obtained from the test matrix. Included in this table are the percent fiber volume in the composite coupons, the percent weight gain due to moisture absorption and the failure modes from each test. For each row (or test), the strength, modulus, shear strength and fracture toughness properties represent averages of five specimens.

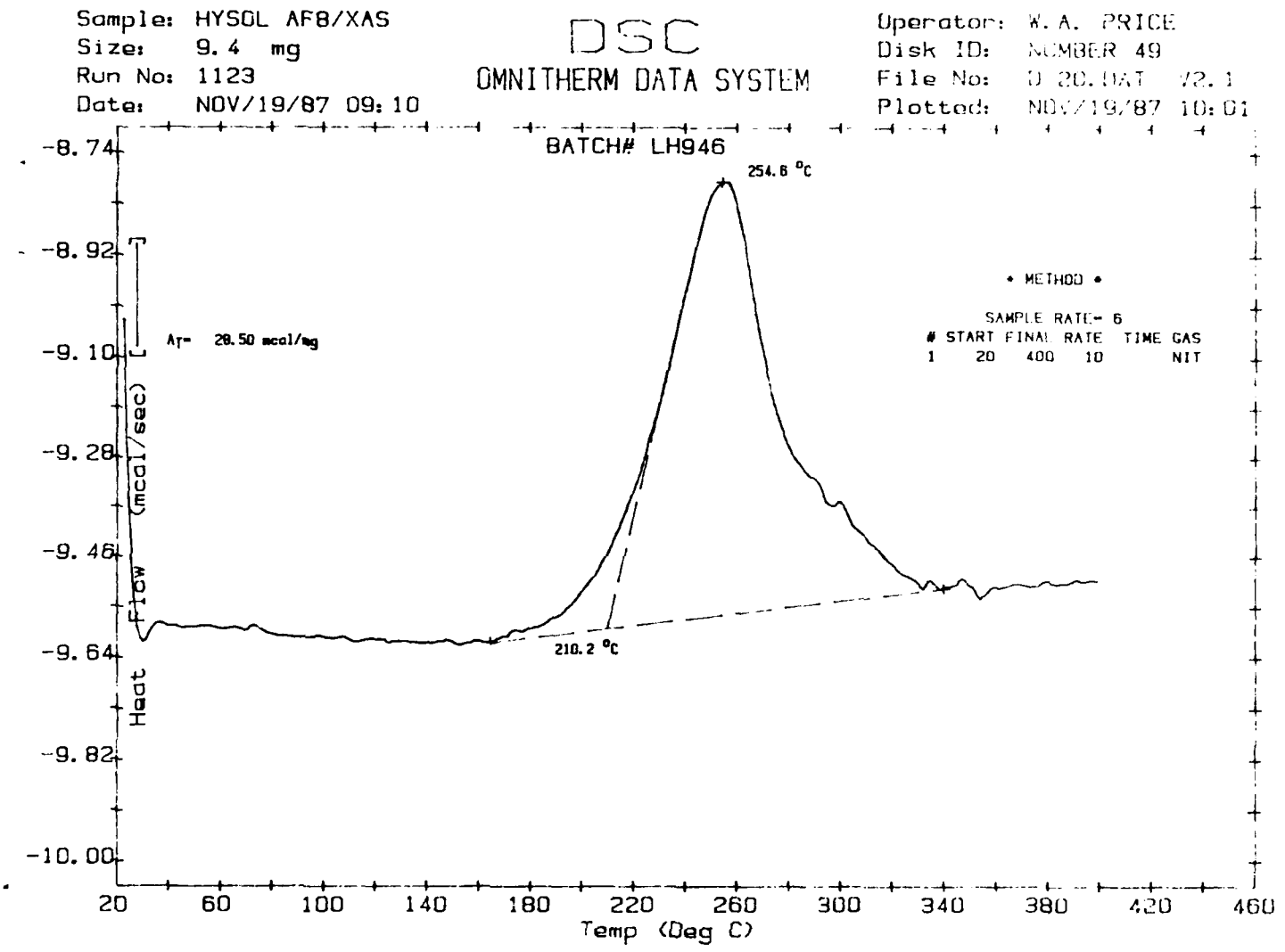


Figure 9 Differential Scanning Calorimeter Trace of XAS/AF-8 Prepreg.

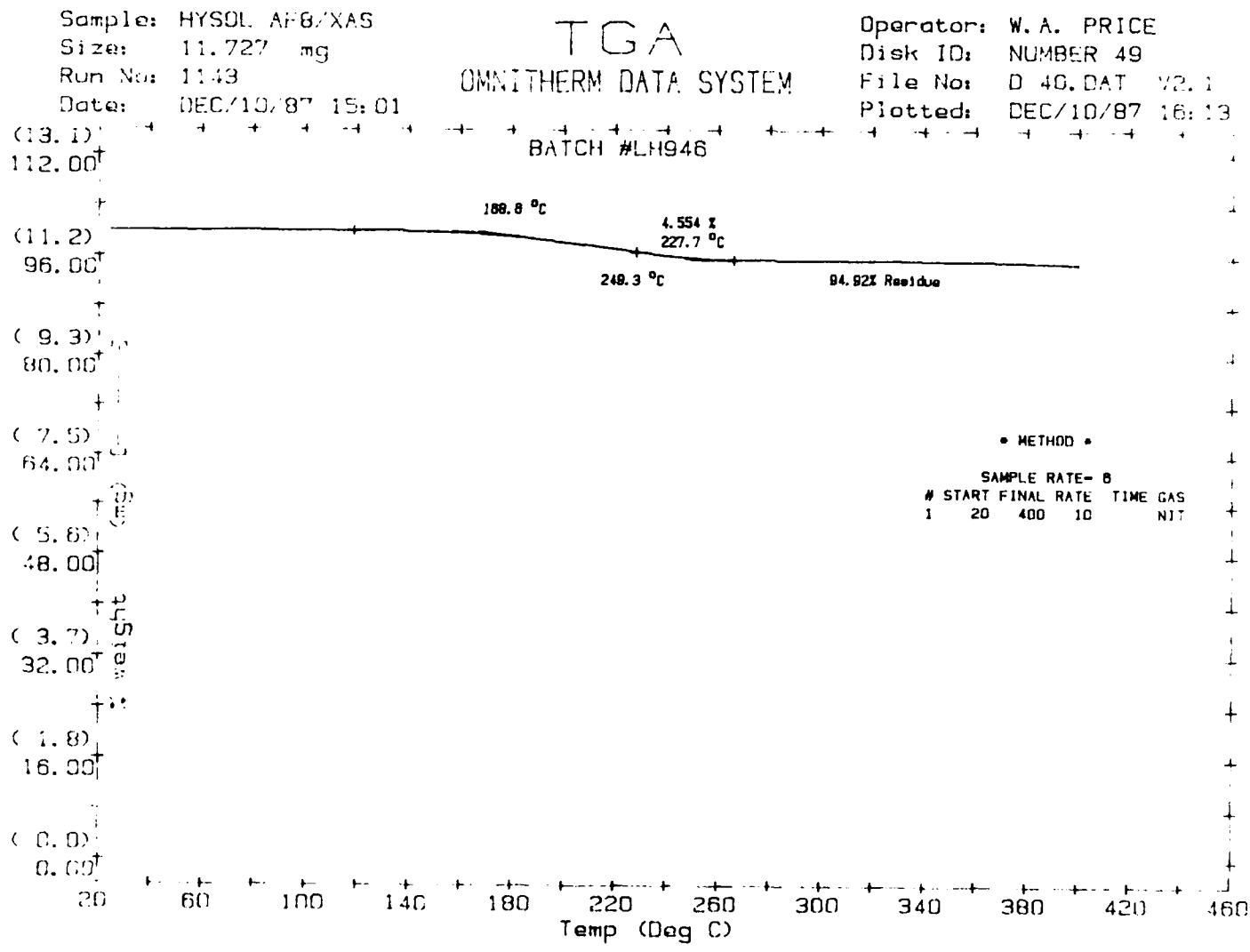


Figure 10 Thermogravimetric Analysis of XAS/AF-8 Prepreg.

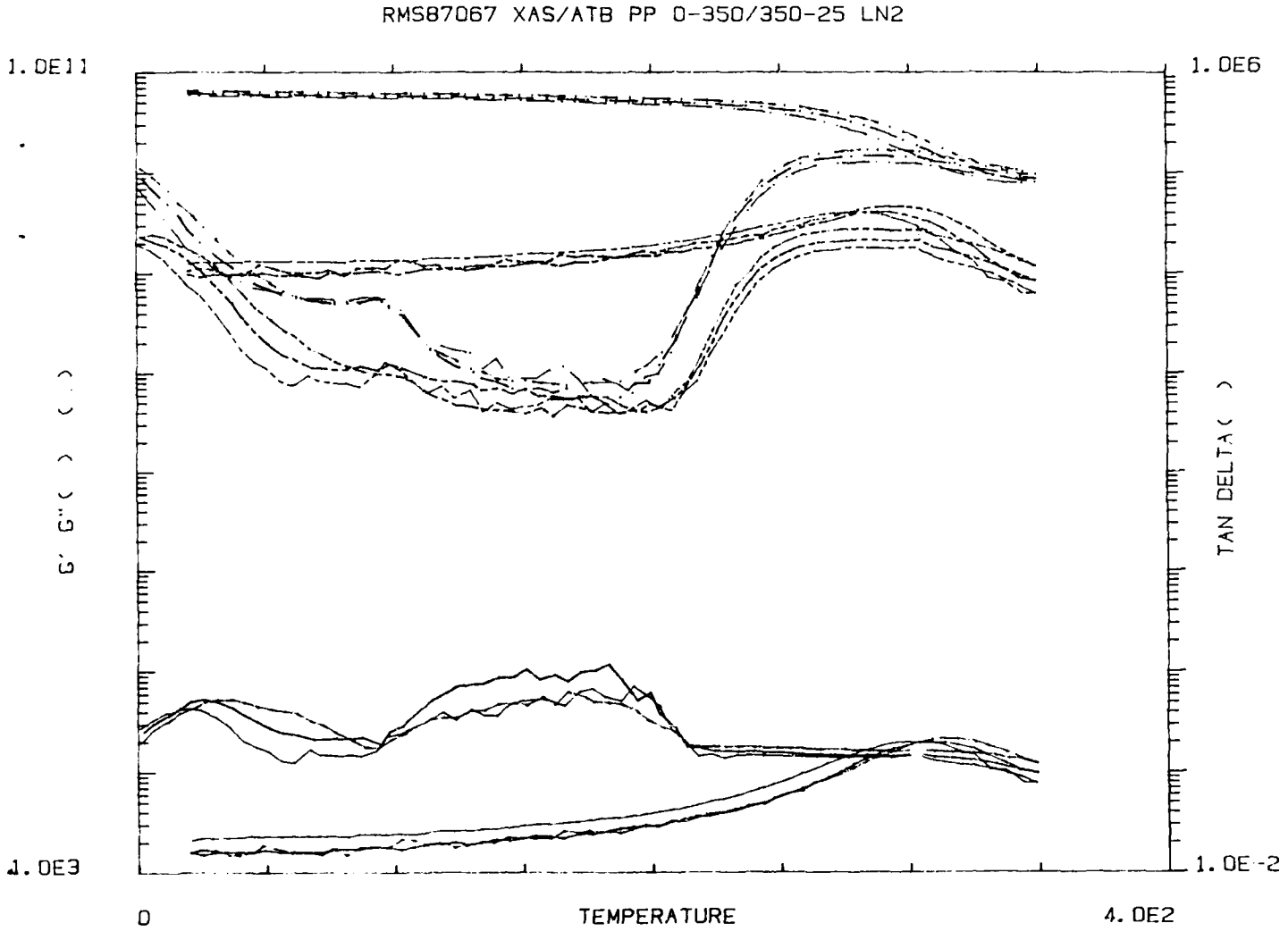


Figure 11 Rheometric Output of XAS/AF-8 Prepreg.

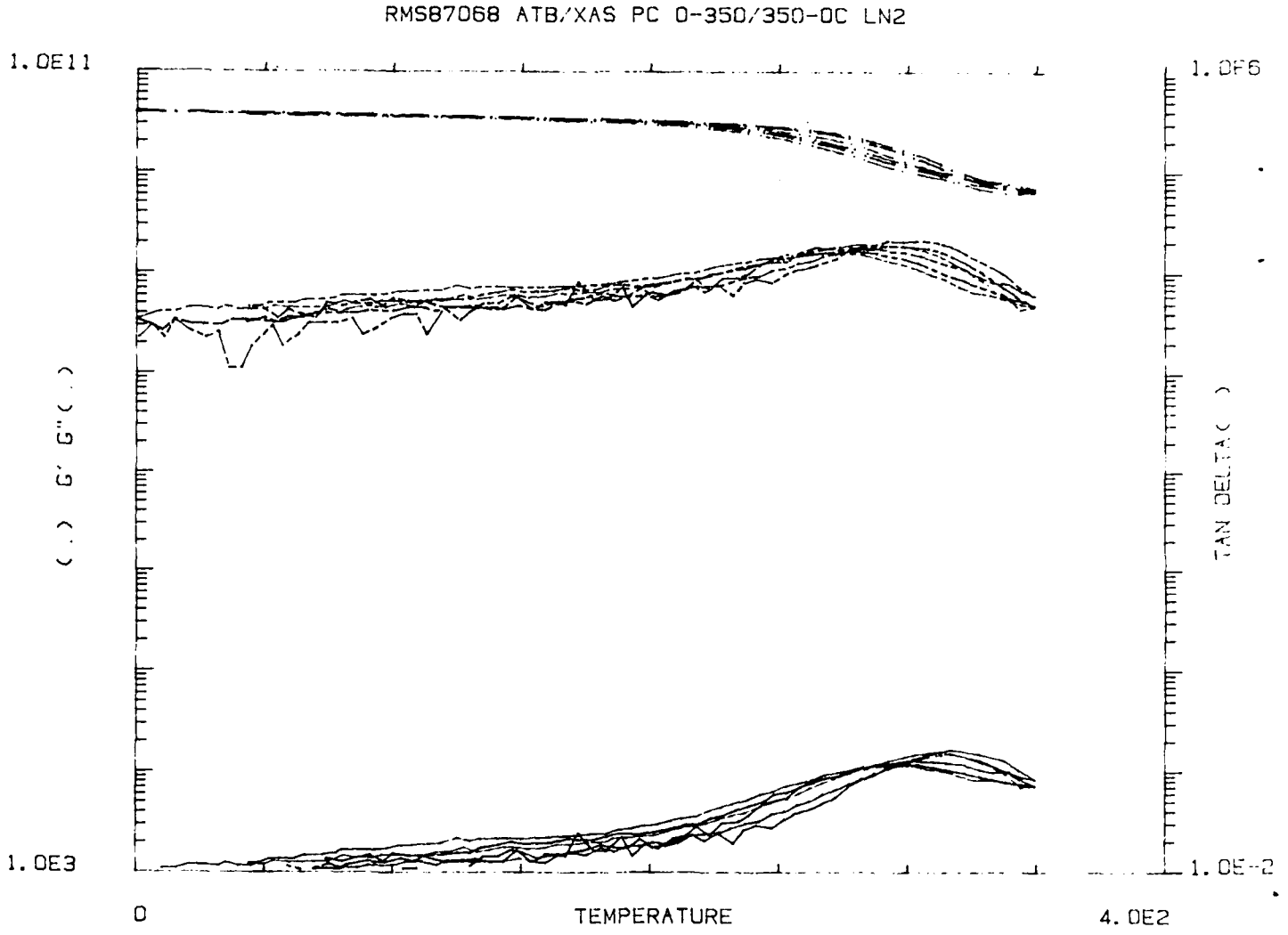
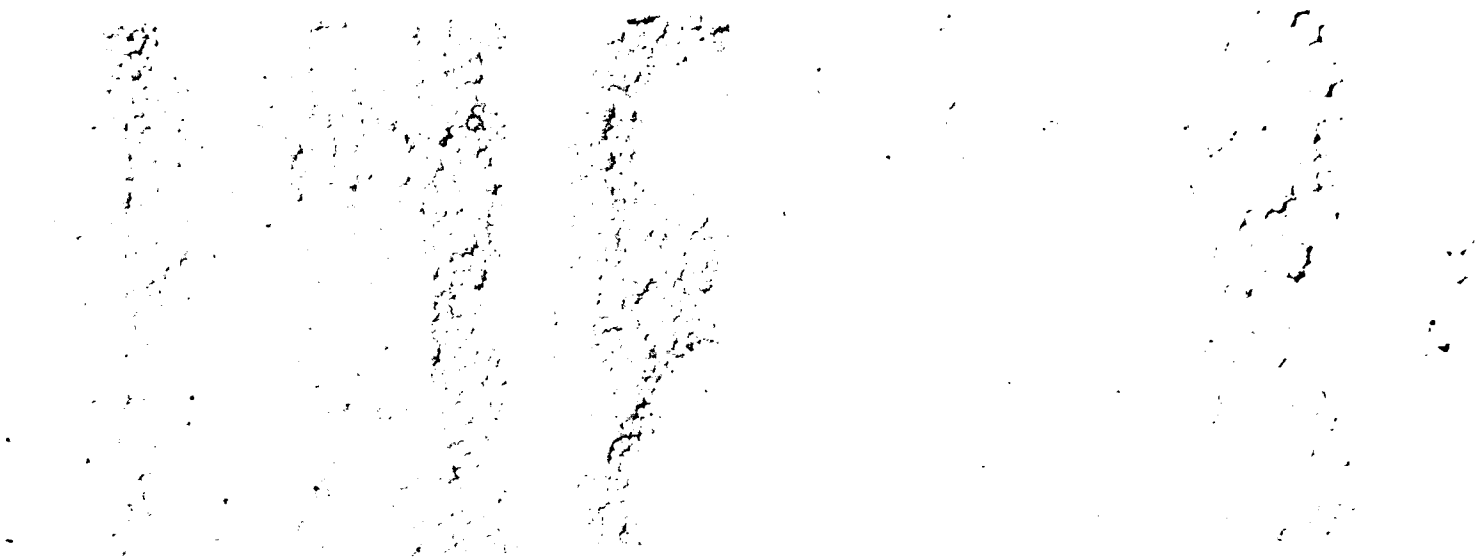


Figure 12 Rheometric Output of As-cured XAS/AF-8 Laminate.



150 x 300 x

Figure 13 Photomicrographs of XAS/AF-8 Panel #5.

Table 4 *Density And Fiber Volume Data For* XAS/AF-8 Panels.

Panel Number	Specific Gravity of Specimen	Specific Gravity of Fiber	Specific Gravity of Matrix	Percent Fiber of Specimen (by volume)	Percent Matrix of Specimen (by volume)
1	1.60	1.79	1.21	63	37
2	1.59	1.79	1.21	63	37
3	1.59	1.79	1.21	63	37
4	1.59	1.79	1.21	62	38
5.a	1.57	1.79	1.21	59	41
5.b	1.56	1.79	1.21	58	42
6	1.59	1.79	1.21	62	38

A plot of the average specimen weight gain versus time for the three batches of moisture-aged specimens is shown in Figure 14. Each batch of specimens achieved 100% saturation in approximately 40 days.

Table 5
Completed Mechanical Test Matrix for XAS/AF-8 Composite

Test	Percent Fiber by Volume	Temperature (°F) 74	250	350	Moisture Gain (Percent)	Strength (Ksi)	Modulus (Msi)	ILS Strength (Ksi)	Failure Mode	Comments
0°-4 Pt. Flex	63	Dry				220	16.0		[1]	
	"		Dry			196	16.6		"	
	"			Dry		160	16.8		"	
	"	Wet			0.75	210	9.1		"	
	"		Wet		0.75	166	8.8		"	
	"			Wet	0.75	119	7.8		"	
0°-3 Pt. Flex	"	Dry				234	15.3		[2]	
	"		Dry			197	14.8		"	
	"			Dry		169	15.1		"	
	"	Wet			0.75	229	15.3		"	
	"		Wet		0.75	199	15.6		"	
	"			Wet	0.75	142	14.7		"	
0°-4 Pt. Shear	"	Dry						12.2	[3]	
	"		Dry					9.9	"	
	"			Dry				7.8	"	
	"	Wet			0.75			10.0	[4]	
	"		Wet		0.75			10.3	"	
	"			Wet	0.75			5.4	"	
0°-SBS	"	Dry						15.4	"	
	"		Dry					12.3	"	
	"			Dry				9.2	"	
	"	Wet			0.63			13.0	"	
	"		Wet		0.63			9.2	"	
	"			Wet	0.63			6.6	"	
90°-4 Pt. Flex	"	Dry				10.0	1.5		[5]	
	"	Wet			0.83	7.1	1.5		"	
Mode II	58	Dry							[6]	[A]
0°-Tension	63	Dry				292	19.3		[7]	[B]
+/-45° Tension	63	Dry				9.3	0.71		[8]	[C]
	63			Dry		9.4	0.47		"	[D]
	62	Wet			0.93	8.9	0.81		"	[E]
	62			Wet	0.95	7.4	0.35		"	[F]
Edge Delam.	63	Dry				41.6	6.1		[9]	[G]
Mode I	58	Dry							[10]	[H]

[1] Mixed Mode: Compression & Interlaminar Shear
[2] Compression
[3] Interlaminar Shear
[4] Compression Buckling
[5] Tension
[6] Mode II Delamination
[7] Complete Splintering of test coupon
[8] Inplane Shear
[9] Mixed Mode: Modes I,II Delamination
[10] Mode I Delamination

[A] Fracture Toughness = 6.53 in-lb/in^2 (based on the compliance reduction method)
[B] Strain to Failure = 0.014 in./in.
[C] Strain to Failure = 0.034 in./in.
[D] Strain to Failure = 0.027 in./in.
[E] Strain to Failure = 0.033 in./in.
[F] Strain to Failure = 0.033 in./in.
[G] Stress at Delamination = 18.0 Ksi
Strain to Failure = 0.011 in./in.
[H] Fracture Toughness = 2.22 in-lb/in^2 (based on the empirical reduction method)

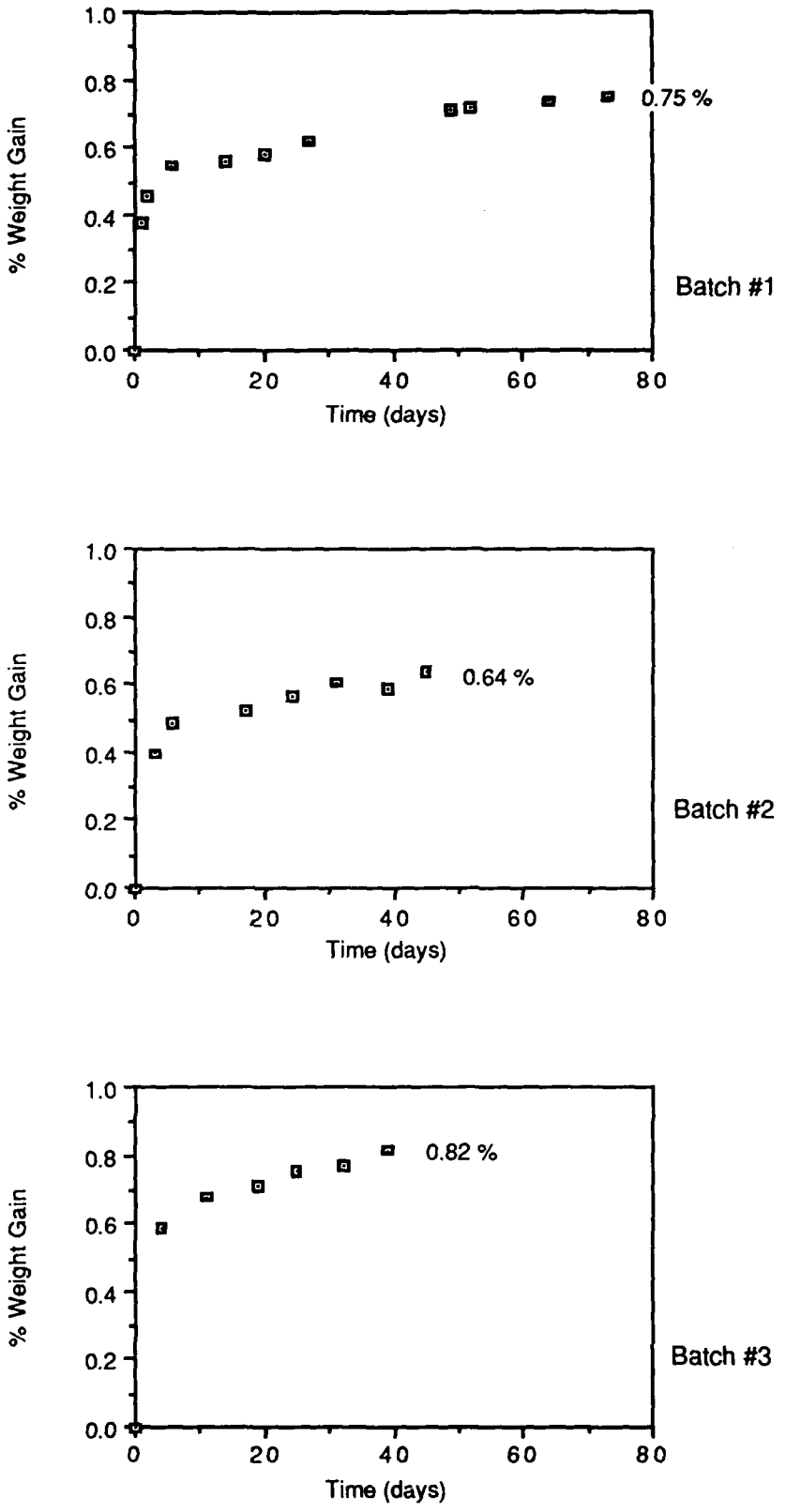


Figure 14 Moisture Weight Gain Versus Time For Three Batches of XAS/AF-8.

V. DISCUSSION

A. Physical Test Data

The relatively low fiber volume of panel #5 can be attributed to the panel's thickness. Panel #5, being 24 plies thick, was autoclave cured with the same number of bleeder plies used on the rest of the panels. Thus, some excess resin remained in Panel #5 as it cured, causing its fiber volume fraction to be lower than expected.

Consider the average percent weight gains for the three moisture-aged batches. Table 6 compares the typical specimen size of each batch with the final average percent weight gain of that batch. As the specimen size increases, the percent weight gain also increases, suggesting that moisture absorption is a function of the specimen geometry.

Table 6 Comparison of Specimen Size With Average Percent Water Absorption for XAS/AF-8 Specimens.

Batch Number	Nominal Coupon Geometry Length (in.)	Width (in.)	Thickness (in.)	Average Percent Weight Gain @ 100% Saturation
2	1.0	0.5	0.07	0.64
1	3.0	0.5	0.07	0.75
3	9.0	1.0	0.05	0.82

B. Mechanical Test Data

The hot/wet data of Table 5 is reproduced graphically in Figures 15 through 19. Both strength and modulus are plotted as a function of temperature and percent water weight gain. These figures show tremendous reduction in properties from room temperature/dry conditions to 350°F/100% H_2O saturated conditions for almost every parameter investigated. Thus, the manufacturer's claim of resilient hot/wet properties is questioned.

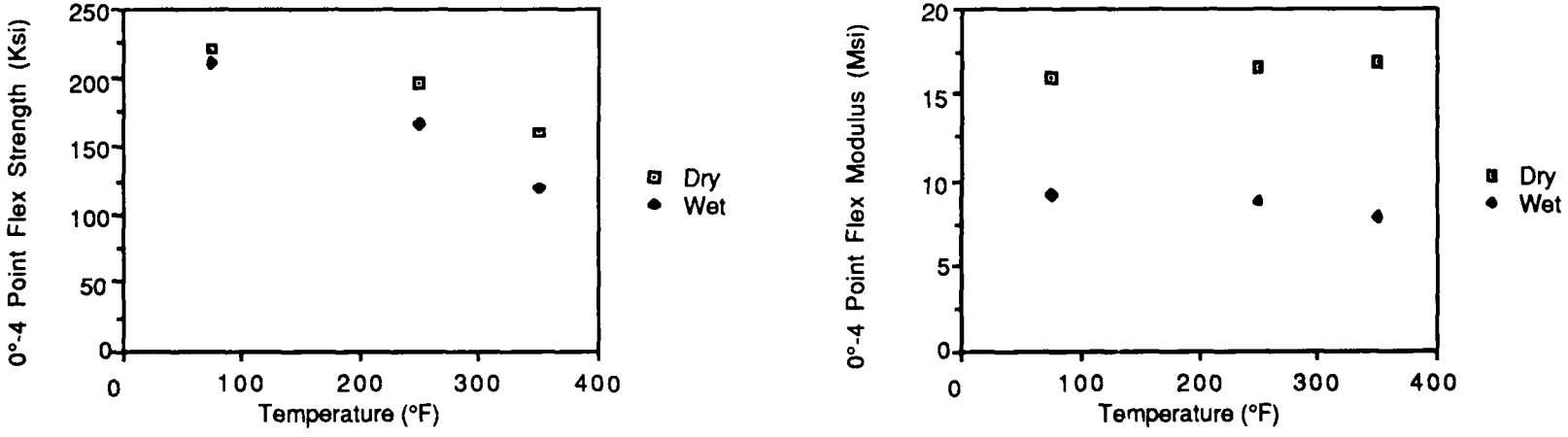


Figure 15 XAS/AF-8 0°-4 Point Flex Hot/Wet Data.

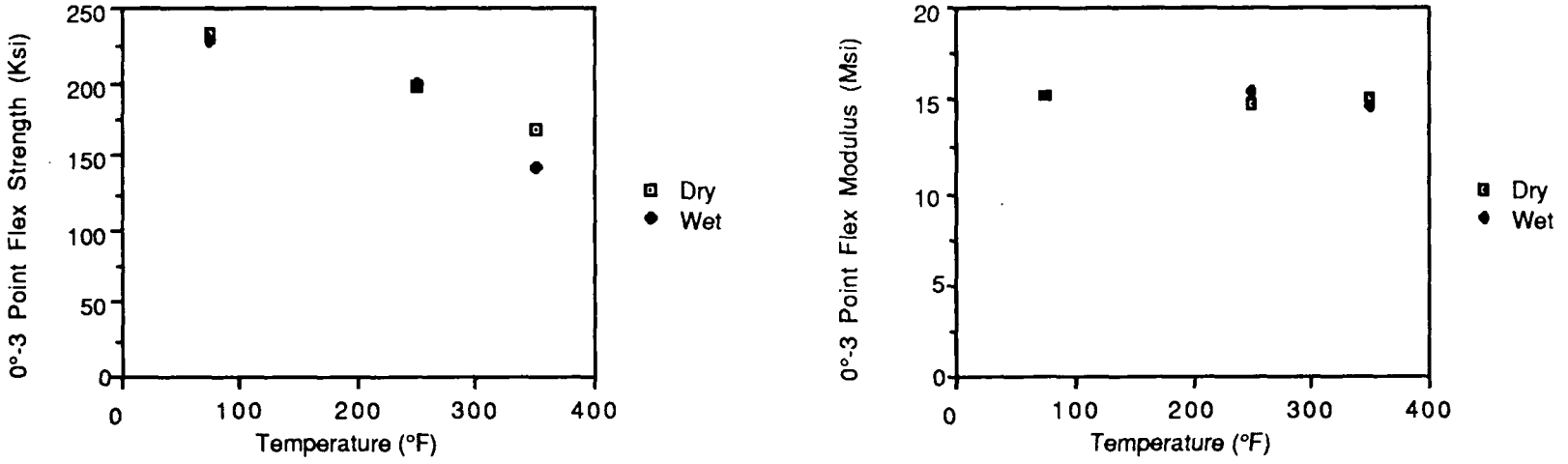


Figure 16 XAS/AF-8 0°-3 Point Flex Hot/Wet Data.

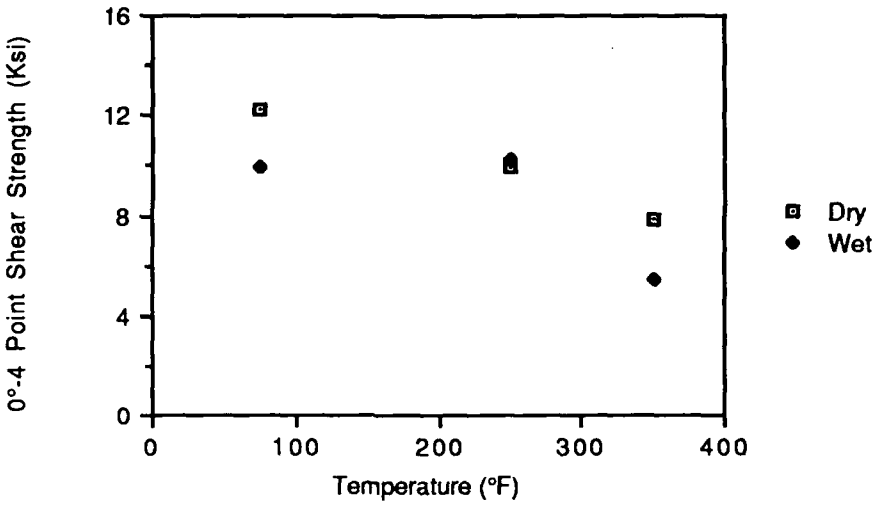


Figure 17 XAS/AF-8 0°-4 Point Shear Hot/Wet Data.

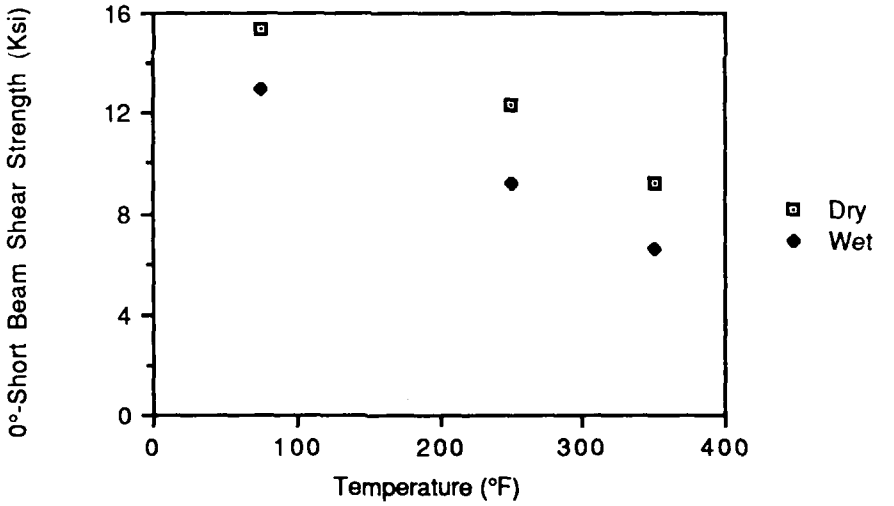


Figure 18 XAS/AF-8 0°-Short Beam Shear Hot/Wet Data.

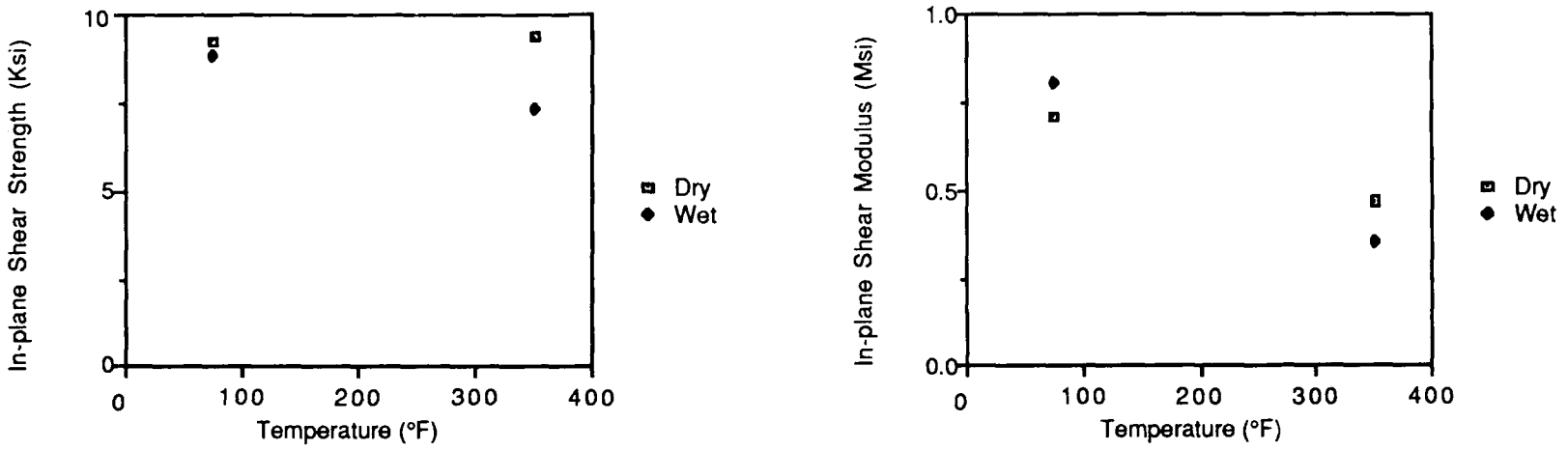


Figure 19 XAS/AF-8 In-plane Shear Hot/Wet Data.

C. Comparison to Manufacturer's Data

Of the manufacturer's mechanical data reported on the XAS/AF-8 system, only a fraction overlaps with and is, thus, comparable to the data generated under this research effort. That portion of the manufacturer's data is reported in Table 7 [6]. Much of the Hysol mechanical data are higher than that recorded in-house. Hysol's tensile data, flex data, in-plane shear strength, and delamination strength are all higher than our data. The interlaminar shear data reported by Hysol are approximately equal to the in-house data, perhaps slightly lower at the wet conditions. We report a higher value for Mode I as compared to Hysol, but this may be due to fiber bridging. Overall, the Hysol values appear higher than documented here. Whether or not this difference is significant is as yet undetermined. The higher values are not the result of an unusually high fiber volume fraction because Hysol reports V_f= 61 ±1%.

Table 7 A Portion of Hysol's XAS/AF-8 Mechanical Data.

Test	Temperature (°F)			Strength (Ksi)	Modulus (Msi)	ILS Strength (Ksi)	Fracture Toughness (in-lb/in^2)
	74	250	350				
0° Flex	Dry			258	17.5		
			Dry	219	17.7		
	Wet			245	17.6		
			Wet	146	16.9		
0° IL Shear	Dry					14.2	
			Dry			8.0	
	Wet					15.4	
			Wet			6.5	
90°-4 Pt. Flex	Dry			11.6			
0°-Tension	Dry			322	20.8		
+/-45° Tension			Wet	17.0	0.35		
Edge Delam.	Dry			23.2*			
Mode I	Dry						1.8

* Stress at delamination

The Hysol final technical report states that their moisture-aged coupons gained, on the average, only 0.04% in weight over the first 7 days. Contrast this value to an average value of 0.54% reported by our research (see Figure 14). Perhaps it is this increase in moisture absorption which is responsible for the decrease in hot/wet

properties. There is no reasonable postulation at this time why our coupons gained more water than Hysol's coupons.

D. Comparison to Alternative Toughened AT System

Table 8 Completed Mechanical Test Matrix For T-300/CATB-44

Test	Percent Fiber by Volume	Temperature (°F)			Moisture Gain (Percent)	Strength (Ksi)	Modulus (Msi)	ILS Strength (Ksi)	Fracture Toughness (in-lb/in^2)
		74	250	350					
0°-4 Pt. Flex	61	Dry				247	17.5		
	"			Dry		195	17.1		
	"	Wet			0.93	222	16.7		
	"			Wet	0.93	128	16.5		
0°-3 Pt. Flex	"	Dry				234	14.5		
	"			Dry		203	15.2		
	"	Wet			0.88	240	19.7		
	"			Wet	0.88	166	7.6		
0°-4 Pt. Shear	"	Dry						12.4	
	65			Dry				8.8	
	61	Wet			1.03			11.7	
	65			Wet	1.03			5.3	
0°-SBS	61	Dry						13.9	
	"			Dry				10.7	
	"	Wet			1.05			13.3	
	"			Wet	1.05			7.3	
90°-4 Pt. Flex	61	Dry				8.7	1.5		
	61	Wet			0.91	6.9	1.5		
Mode II		Dry							1.64
0°-Tension	61	Dry				241	17.3		
+/-45° Tension	64	Dry				10.1	0.95		
	64			Dry		10.9	0.93		
	64			Wet	1.48	6.8	0.58		
Edge Delam.		Dry				19.4*			
Mode I		Dry							0.59

* Stress at delamination

Table 8 shows the mechanical data reported by Curliss[7] on American Cyanamid's "toughened" acetylene-terminated resin system. The mechanical tests which are most important in quantifying the materials hot/wet and toughness properties are the flexure, shear, ±45°-tension and Mode I tests. In comparing the two data sets, it appears as though the flex and shear properties show similar strength and modulus reductions as a function of increasing temperature and water gain. The Mode I data from the

XAS/AF-8 system is much higher (by a factor of four) than that of the T-300/CATB-44 system. Again, the unusually high Mode I value may be the result of fiber bridging. However, this piece of data initially suggests that Hysol's toughening system is more effective than American Cyanamid's system.

VI. CONCLUSIONS

The procedures used to fabricate the composite laminates from the XAS/modified ATB prepreg were followed rigorously. Strict standards were followed where applicable. And, consistent procedures were followed where standards had yet to be written. The laminate physical data appeared relatively normal, suggesting that the mechanical data is a valid (but limited) profile of the material's intrinsic properties. Direct comparison of mechanical test data between two materials can be difficult because oftentimes key particulars associated with the test are not reported. The span-to-depth ratio of flexural tests, the failure modes on all tests and percent fiber volume data are all necessary in order to make a reasonable comparison between materials.

The XAS/AF-8 system falls short of its supposedly improved hot/wet properties. However, the material's Mode I fracture toughness remains intact. Contrast this data to previous claims that AT systems are, in general, unmanageable in the lay-up stages, difficult to process and brittle. From all of this it appears as though the toughening process has yet to be optimized. Further investigations looking to optimize the AT's toughness--hot/wet trade-off may wish to concentrate on the problem of high moisture gain.

VII REFERENCES

1. **Browning, C. E.**, The Mechanisms of Elevated Temperature Property Losses In Epoxy Matrix Composites After Exposure To High Humidity Environments, Doctoral Thesis, University of Dayton, 1976.

2. **Hedberg, Frederick L.**, Polymer Chemist, Materials Laboratory, Wright-Patterson Air Force Base, Ohio; In-house memorandum, 1987.

3. **Whitney, J. M., Browning, C. E.**, On Short Beam Shear Tests for Compcsite Materials, Experimental Mechanics, Vol. 25, No. 3, 294-300, September 1985.

4. **Wagner, P. R.**, Use Of The Four Point Flex Test for Examining The Compression Stress-Strain Behavior Of Unidirectional Composites, 34th International SAMPE Symposium, May 8-11, 1989.

5. **Whitney, J. M., Husman, G. E.**, Use of the Flexure Test for Determining Environmental Behavior of Fibrous Composites, Experimental Mechanics, Vol. 18, No. 5, 185-190, May 1978.

6. **Steiner, Paul A., McKillen, John M.**, Dexter Hysol Final Report for Work Performed Under USAF Contract F33615-84-C-5115.

7. **Curliss D. B.**, Physical And Mechanical Properties of American Cyanamid's Acetylene-Terminated Prepreg System: T-300/CATB-44, AFWAL-TR-88-4243.

☆U.S. GOVERNMENT PRINTING OFFICE: 1989 - 748-056/24011